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Indian Standard

METHODS FOR DETERMINATION OF STRENGTH OF INDIGO IN SUBSTANCE

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METHODS FOR DETERMINATION OF STRENGTH OF INDIGO IN SUBSTANCE

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Indian Standard

METHODS FOR DETERMINATION OF STRENGTH OF INDIGO IN SUBSTANCE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 March 1986, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

0.2 Indigo, on account of its fastness to light and washing, beauty of shade and other special properties is predominently used in colouring of textiles. In view of this and of its high price, the accurate estimation of the amount of pure colouring matter present in commercial indigo is very important.

0.3 Synthetic indigo, which now forms the chief source of supply, comes in the form of a fine powder containing 96 to 98 percent pure indigo (indigotine). It is also sold as a thin paste containing 20 percent of dyestuff, or in the form of a concentrated solution of the leuco compound (reduced indigo). Natural indigo is less pure and contains indigo red (indirubine), small quantities of certain brown and yellow colouring-matter, glutinous material and mineral matter (clay and sand).

0.4 The methods given in this standard are based on the oxidation of indigo to isatine by use of a standard solution of potassium permanganate, Disulphonic acid method also gives an approximate estimation of indirubine present in natural indigo and is less accurate. Tetrasulphonic acid method is not as rapid as the disulphonic acid method but is more accurate and is to be preferred when impure indigos are to be estimated. This method also gives exact estimation of indirubine present in natural indigo. With indigo of high purity both the methods agree well. In the case of both methods, however, the presence of starch in the product may lead to erraneous (too low) results. In such cases it is necessary to previously boil the sample with dilute hydrochloric acid (4 percent) for an hour before proceeding to the analysis.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

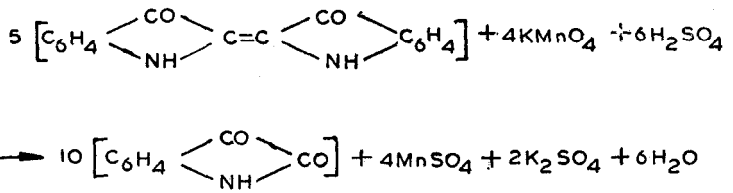
*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes two methods for estimation of pure colouring matter (indigotine) present in the commercially available natural and synthetic indigo in power or paste form or in the form of concentrated solution of leuco compound.

2. PRINCIPLE

2.1 A test sample of commercial indigo is first converted into a disulphonic acid, or into a tetrasulphonic acid to render it soluble in water and is then oxidized by a standard solution of potassium permanganate to form isatin-mono-or-disulphonic acid. The reaction proceeds theoretically according to the equation:



On account of the high dilution or for other unascertained reasons, the reaction does not take place strictly in accordance with the above equation and a factor is therefore used which represents the results obtained experimentally with pure indigotine under the same conditions. This factor varies somewhat with the method of estimation employed.

3. STANDARD INDIGO

3.1 The standard sample of indigo against which the strength of indigo under test is evaluated shall be as agreed to between the buyer and the seller.

4. APPARATUS

4.1 Drying Oven — capable of drying at 110 ± 2°C.

4.2 Weighing Balance — with an accuracy of 1 mg.

4.3 Glass Rod — for stirring purpose.

4.4 Glass Beakers — capacity of 250 ml.

4.5 Water Bath — capable of heating at 70 to 75°C.

4.6 Conical Flasks — 500 ml and 200 ml capacity.

4.7 Titration Flask — 500 ml capacity.

4.8 Burette — graduated in ml.

4.9 Pipette — 25 ml capacity.

4.10 Gooch Crucible — 30 ml capacity.

4.11 Funnel

4.12 Shallow Porcelain Dish — of suitable size.

5. REAGENTS

5.0 Quality of Reagents — Unless otherwise specified, pure reagents shall be employed. Whenever the use of water as reagent is intended; pure distilled water as per IS : 1070-1977* shall be used.

NOTE — Pure reagents shall mean reagents which do not affect the result of test or analysis.

5.1 20 g of White Quartz Sand

5.2 Pure Concentrated Sulphuric Acid — 98 percent (*m/v*).

5.3 Barium Chloride Solution — 10 percent (*m/v*).

5.4 Potassium Permanganate Solution — N/50, prepared by dissolving 0·632 g potassium permanganate per litre of the solution with water.

5.5 Fuming Sulphuric Acid — 20 percent SO_3 for fairly pure indigo and 25 percent SO_3 for crude indigo.

5.6 Potassium Acetate Solution — 450 g/l.

5.7 Ice

5.8 Glacial Acetic Acid

5.9 Hydrochloric Acid Solution — 4 percent (*m/v*).

6. PROCEDURE COMMON TO BOTH METHODS

6.1 Take the required quantity of test specimen (*see* **6.2** *and* **6.3**) and indigo from the test sample selected (*see* **8.3**), in a beaker and boil the test specimen with dilute hydrochloric acid (4 percent, *m/v*) for an hour to remove any starch if present. Extract the boiled specimen with distilled water thoroughly and proceed for estimation as given in **6.2** and **6.3.**

6.2 Disulphonic Acid Method

6.2.1 Weigh accurately 0·5 g of the test specimen and treat as in **6.1.**

*Specification for water for general laboratory use (*first revision*).

6.2.2 Dry it at 110 ± 2°C in the drying oven for 2 to 4 hours till a constant mass is obtained. Take it into a beaker and add to it about 3 g of white quartz sand and 20 ml of pure concentrated sulphuric acid.

6.2.3 Mix the contents with a glass rod and heat in a water bath at a temperature of 70 to 75°C for an hour, the mixture being meanwhile occasionally stirred.

6.2.4 Pour off the sulphuric acid solution from the sand into a 500 ml flask.

6.2.5 Transfer the washing of the remaining sand, glass rod and the beaker with water into the flask.

6.2.6 Dilute the contents of flask (*see* **6.2.5**) to about 400 ml and add 10 ml of a 10 percent solution of barium chloride and make the whole up to 500 ml with water.

NOTE — The barium sulphate formed settles quickly and carries down with it impurities present in the indigo which would otherwise mask the end point of the titration.

6.2.7 After keeping the contents of flask as in **6.2.6** for half an hour, draw 50 ml of clear solution in the titration flask, dilute it with 300 ml of distilled water and titrate the contents with N/50 potassium permanganate solution. Note the end point when the solution has a pale yellow or orange colour free from any bluish or greenish tint. Note the volume of potassium permanganate used to reach end point.

6.2.8 Calculate the quantity of indigotine present in the test sample by factor: 1 ml of N/50 $KMnO_4$ = 0·001 47 g indigotine.

6.2.9 Repeat the procedure from **6.2.1** to **6.2.8** for the standard indigo (*see* **3.1**).

6.2.10 Calculate the percentage strength of indigo under test by the following formula:

$$S = \frac{V_2}{V_1} \times 100$$

where

S = percentage strength of indigo under test,

V_1 = volume of N/50 $KMnO_4$ used in titration of standard indigo, and

V_2 = volume of N/50 $KMnO_4$ used in titration of indigo under test.

NOTE 1 — With natural indigo containing more than 1 or 2 percent of indirubine the end point instead of being yellow is orange or red whose tint, however, changes to yellow upon further addition of potassium permanganate. The quantity of indirubine present thus may be calculated approximately as follows:

$$\text{Percentage of indirubine on pure indigotine basis} = \frac{V_3 - V_4}{V_3} \times 100$$

where

V_3 = volume of potassium permanganate used when the end point is yellow, and

V_4 = volume of potassium permanganate used when the end point is orange or red.

NOTE 2 — For more exact estimation of indirubine, tetrasulphonic acid method should be used.

6.3 Tetrasulphonic Acid Method

6.3.1 Weigh accurately 1 g of the test specimen and treat as given in **6.1**. Take it into a beaker and dry in the drying oven at 110 ± 2°C for 2 to 4 hours till a constant mass is obtained. Add to it 3 g of white quartz sand and 5 ml of fuming sulphuric acid (20 percent SO_3 for fairly pure indigo or 25 percent SO_3 for crude indigo). Stir the mixture with a glass rod, cover with a watch glass and heat in the water bath for 45 minutes at 96 to 98°C stirring at intervals.

6.3.2 Then cool the contents to room temperature, transfer the clear sulphonic acid solution to a 500 ml flask containing a little water and add to it the washings of the beaker, sand and the glass rod. Make the volume up to 500 ml with water.

6.3.3 Take 100 ml of a solution obtained in **6.3.2** into a conical flask and add to it 100 ml of a solution of potassium acetate (450 g/l). Heat the mixture just to boiling point in order to dissolve the precipitate first formed. Then cool it at once in running water and finally allow to stand for an hour upon ice.

NOTE — If the acetate solution is boiled for any length of time, there is a liability for destruction of indigo to occur.

6.3.4 Collect the potassium tetrasulphonate which separates out upon cooling as a crystalline precipitate, in a Gooch crucible and wash it with an ice cold solution containing 225 g/l of potassium acetate and 125 g/l of glacial acetic acid.

NOTE — If the indigo is pure, the filterate is nearly colourless, or at least has only a pale blue tint. With impure indigo the colour of the filterate will depend upon the character of impurities, thus if indirubine is present, a red filterate is obtained.

6.3.5 Place the Gooch crucible containing the potassium tetrasulphonate in a funnel resting in the neck of a 200 ml flask and wash the contents with hot water, adding any precipitate which may have remained adhering to conical flask. Make the contents of the flask up to 200 ml.

6.3.6 Transfer 200 ml of the solution as obtained in **6.3.5** to a shallow porcelain dish. Add 0·5 ml of pure concentrated sulphuric acid, dilute with 80 ml of water and titerate with a potassium permanganate solution (N/50) till the end point is reached (*see* **6.2.7** *and* Note 1 under **6.2.10**).

6.3.7 Calculate the quantity of indigotine present by the factor: 1 ml of N/50 $KMnO_4$ = 0·001 47 g indigotine.

6.3.8 Repeat the procedure from **6.3.1** to **6.3.7** for the standard indigo (*see* **3.1**).

6.3.9 Calculate the percentage strength of indigo as follows:

$$\text{Percentage strength} = \frac{V_6}{V_5} \times 100$$

where

V_5 = volume of N/50 $KMnO_4$ used to reach the end point for the standard indigo (*see* **3.1**), and

V_6 = volume of N/50 $KMnO_4$ used to reach the end point for the indigo under test.

6.3.10 Percentage of indirubine can be calculated by noting the volumes of N/50 $KMnO_4$ used when the end point is orange red and yellow (*see* Note 1 *under* **6.2.10**) by the following formula:

$$\text{Percentage of indirubine on pure indigotine basis} = \frac{V_7 - V_8}{V_7} \times 100$$

where

V_7 = volume of N/50 $KMnO_4$ used when the end point for indigo under test is yellow, and

V_8 = volume of N/50 $KMnO_4$ used when the end point for indigo under test is orange or red.

7. REPORT

7.0 The report shall indicate the following.

7.1 Percentage strength of indigo as obtained in **6.2.10** and **6.3.9**.

7.2 Percentage of indirubine present in the indigo on the basis of pure indigotine as obtained in **6.2.10** (Note 1) and in **6.3.10**.

8. SAMPLING

8.1 Lot—The quantity of commercial indigo of similar form and composition delivered to a buyer against one despatch note shall constitute a lot.

8.2 Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected at random from a lot shall be as follows:

Lot Size	*Sample Size*
Up to 8	2
9 to 15	3
16 to 24	4
25 and above	5

8.3 If the indigo is in powder form, draw randomly small quantities of dyestuff by a suitable sampling instrument from at least five different parts of each selected container and mix them thoroughly to get a test sample of about 20 g. If the indigo is in paste or concentrated solution form, stir the contents of each container selected, thoroughly with a wooden stick so as to homogenise the contents. Then draw randomly small quantities of dyestuff by a suitable sampling instrument from at least five different parts of each selected container and mix them thoroughly by stirring with a glass rod so as to get a test sample of about 20 g.

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